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(71) Applicant : **OWENS-ILLINOIS CLOSURE INC.**
One SeaGate
Toledo Ohio 43666 (US)

(72) Inventor : **Bayer, John W.**
10492 Bayer Road
Perrysburg, Ohio 43551 (US)

(74) Representative : **Votier, Sidney David et al**
CARPMAELS & RANSFORD
43, Bloomsbury Square
London WC1A 2RA (GB)

(54) **Closure construction for hot fill and retort applications.**

(57) A plastic closure for sealing containers which have been filled with contents that are hot or which are to be retorted. The closure is made of thermosetting or thermoplastic material. The closure includes a base wall and a peripheral skirt. The skirt is formed for engaging a container. The base wall of the closure having an inner surface with a liner thereon. A reactive hot melt adhesive bonds the liner to the inner surface. The reactive hot melt adhesive is cross-linkable such that after the liner is applied and the adhesive cures, the adhesive bonds the liner to the inner surface of the base wall of the closure such that the liner will withstand and resist deformation under vacuum caused by cooling of the hot contents in a container or caused by retorting the contents of a container and subsequent cooling. The reactive hot melt adhesive may be a cross-linkable adhesive selected from the group consisting of polyurethane and silicone. The liner being adhered may be made of ethylene, polypropylene, α-olefin copolymers, i.e. ethylene-octane, propylene-ethylene or butylene-ethylene and SBR rubber.

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This invention relates to plastic closures utilized for hot fill and retort applications.

Background and Summary of the Invention

Plastic closures with sealing liners for glass and plastic containers may be classified as follows:

1. Adhesives for liner/glass interface - These include adhesives applied to the side of the liner which under heat, bonds to the container finish to generally impart tamper indication. Patents in this category are 4,684,554 and 4,778,698.

2. Laminating Adhesives - Adhesives in this type consist of adhesives for bonding two or more films together to form a liner composition. They do not bond the liner to the closure shell interface. Patent 4,961,986 mentions use of a water borne urethane utilized as a laminating adhesive. Other patents utilize thermoplastic adhesives such as 4,961,986, 2,263,693, 3,866,845, 4,818,577 and 4,930,646.

3. Thermoplastic gaskets - Use thermoplastic hot melts as the sole gasketing compound generally applied as an annular ring. For example a fully cured thermoplastic urethane elastomer is applied in a crown closure as a completely reacted polymer. Patents in this category are 4,032,492, 4,085,186, 4,336,011, 4,852,754, 4,988,467 and 4,968,514.

4. Adhesives - generally are directed towards some other area such as closure construction. Patents that mention the use of adhesives but do not define the chemical makeup of the adhesive such as 3,976,217 and 4,576,297.

5. Liner to shell Adhesives - Patents which describe a composition of the adhesives for bonding a liner to a closure shell, such as lacquer or sizings applied from solution to a metal shell or include a curing step to bond the liners by heat or pressure to the shell. See for example patents 2,976,200, 3,496,060, 4,151,924 and 4,280,864.

Closures with mechanically locked liners for hot fill and retort applications consist of a plastisol is mechanically held in a specially designed closure. The plastisol is applied and heat cured. Such a closure requires extensive capital equipment, plant floor space and labor to produce.

Among the objectives of the present invention are to provide a closure which effectively can be used for hot fill and retortable applications such as food products, wherein there is a minimal modification required in making the closure with a liner; wherein there is no need for post-curing equipment; and wherein there is no additional space or labor above that needed for conventional lining operations.

In accordance with the invention, a plastic closure is provided for sealing containers which have been filled with contents that are hot or which are to be retorted. The closure is made of thermosetting or thermoplastic material. The closure includes a base wall and a peripheral skirt. The skirt is formed for engaging the finish of a container. The base wall of the closure has an inner surface with a liner thereon. A reactive hot melt adhesive bonds the liner to the inner surface. The reactive hot melt adhesive is cross-linkable such that after the liner is applied and the adhesive cures, the adhesive bonds the liner to the inner surface of the base wall of the closure such that the liner will withstand and resist deformation under vacuum caused by cooling of the hot contents in a container or caused by retorting the contents of a container and subsequent cooling. The reactive hot melt adhesive may be a cross-linkable adhesive selected from the group consisting of polyurethane and silicone. The liner being adhered may be made of ethylene, polypropylene, α-olefin copolymers, i.e. ethylene-octene, propylene-ethylene or butylene-ethylene.

Description

In accordance with the invention, the closure is made of thermosetting or thermoplastic material. The closure includes a base wall and a peripheral wall. The closure includes means such as threads on the inner surface of the peripheral wall for engaging container.

The plastic closure is intended to be used for sealing containers which have been filled with contents that are hot or which are to be retorted. A reactive hot melt adhesive bonds the liner to the inner surface of the base wall. The reactive hot melt adhesive is of the type which is cross-linkable after the liner is applied and upon curing bonds the liner to the inner surface of the base wall of the closure. As a result, the liner withstands and resists deformation under vacuum caused by cooling of the hot contents in a container or caused by retorting the contents of a container and subsequent cooling.

Currently plastic closure liners for hot fill applications where vacuum is generated have been limited to an annular ring applied plastisol which is mechanically locked in place to prevent the gasket from being drawn into the container as vacuum is generated. Disc liners (gaskets) have not been utilized for this reason, but could be if adhered to the shell. To date, the current hot melt types of materials are too soft at the food processing temperatures causing the liner to also be drawn into the container during the cool down and vacuum formation.

Plastisol lined closures are expensive due to the specially designed shell, complex application equipment, and the need for an expensive and long curing oven.

In one form, the hot melt adhesive of the present invention is preferably polyurethane. In another form, the adhesive comprises a reactive silicone.

It has been found that reactive hot melt adhesives can overcome the deficiencies of normal hot melts. The reactive hot melt adhesives can be applied by standard equipment but after lining react to become cross linked thus becoming immobilized from flow at higher temperatures after a period of curing at room temperature. After adequate cure the liner bonded to the closure shell will withstand and resist the deformation of the liner under vacuum and not be sucked into the container and lose its seal.

Another disadvantage of plastisol lined plastic closures has been the oxygen permeation through the top panel of the closure which limits their use to foods that are not highly susceptible to oxygen degradation. The present invention allows latitude in that liner constructions containing aluminum foil or other barrier materials can be utilized to minimize oxygen ingress through the closure panel.

Phenolic and polypropylene closure shells were lined on production equipment. The liners tried in both shells were F-217 polyethylene liner and Norton Teflon faced urethane liner. National Starch 70-7254 hot melt adhesive was used as the cross-linkable adhesive. The liners could be stripped out by hand by cohesive failure mechanism of the adhesive until about two days after lining showing that the adhesive had cured due to atmospheric moisture. After this time adhesive failure occurred between the F-217 liner and the polypropylene interface. Adhesion was rated poor to medium. In the case of the phenolic shell and the urethane liner, the liner tore when pulled apart by hand. Since this time, the inside of the polypropylene shells have been either corona or flame treated with excellent bonding to the polypropylene occurring with the 70-7254 adhesive. Again after two days curing at atmospheric conditions the liner would tear when pulled apart.

The closures lined were applied to 16 oz. 38mm glass containers filled with boiling water and headspaced to 3/8". The containers were allowed to cool overnight. Vacuum could not be determined by a vacuum gage since the closures were brittle and would crack on insertion of the needle but on opening a hiss was apparent which indicated that vacuum was present. There was no distortion of the closure due to the temperatures involved in the capping operation.

As a specific example the closure is constructed as follows:

1. Shell - polypropylene.
2. Liner - foamed polyethylene material such as F-217, vinyl with modification dictated by end use, i.e., foil, solid layers, etc.
3. Adhesive - the reactive hot melt adhesive comprises a reactive polyurethane. Such an adhesive comprises National Starch 6902-1, National Starch 70-7254, Fuller M-3082 or Bostic 9601, National Starch 6902-1 comprises urethane, made by National Starch and chemical Corp., Bridgewater, New Jersey, National Starch 70-7254 comprises urethane made by National Starch and Chemical Corp., Bridgewater, New Jersey, Fuller M-3082 comprises urethane, made by H. B. Fuller Co., St. Paul, Minnesota. Bostic 9601 comprises a urethane made by Bostic, Middleton, Massachusetts. Another reactive adhesive comprises silicone adhesive. Such an adhesive is R116 silicone adhesive made by General Electric Silicones, Waterford, New York.

The above are examples since other closure shell constructions, liner combinations and modifications of the adhesives can be substituted.

The reactive adhesive can be applied by current or modified application equipment. The reactive adhesive should be uniform in thickness and cover 75-80% of the interior surface of the panel. The liner can be cut and inserted with present equipment. Closures with the liner applied require moisture to cure so require about one week of time in +50% RH atmosphere before use.

A preferred liner composition comprises olefin copolymers which are produced from new catalyst technology which can tailor molecular weight, molecular weight distribution, crystallinity and the distribution of comonomers. Through this technology, specific physical and chemical properties can be achieved in the reactor, which can be utilized for liner applications. In comparison, materials such as thermoplastic olefins (TPO) which are currently used today, achieve their physical properties through the blending of two or more polymeric compounds and a variety of additives which may contribute to contamination of the product. Hot fill packaging is one application being considered for the new liner material.

In one example, a propylene-ethylene copolymer liner formulation was formed and tested which was designated VC-1. The formula consisted of Himont 11223-67-1 and PMS 2B38947NMBD1 in a 10:1 ratio, this formula contained 1% oleamide. Himont 11223-67-1 comprises propylene-ethylene. PMS 2B 38947NMBD1 comprises 89% linear low density polyethylene, 11% oleamide and 0.001% phthalocyanine pigment made by PMS Inc. Norwalk, Ohio. The components were mixed together on a roll mill and then 0.050 inch thick sheets were pressed out using a hot press. Die cut disks were then punched out from these sheets to form the liners. These

liners were adhesively bonded to the closures using the reactive hot melt adhesive. Room temperature removal torques after one month measured 11.6 inch-pound. Hardness was found to be Shore A 94. Oxygen permeability was found to be 0.059 cc(STP) day atm at 73°F and wet conditions.

Another application that has been investigated is for autoclave use. Black phenolic closures were lined with corona treated Himont 11223-67-1 material. This material comprises propylene-ethylene and is made by Hinmont USA Inc., Wilimington, Delaware. The liner was 0.040 inches thick and adhesively bonded to the closure by a hot melt adhesive comprising National Starch 70-7254. The closures were applied to glass bottles with a force of 10 inch-pounds. The closures were autoclaved at 125°C for thirty minutes, for a total of five cycles without loss of adhesion. The closure liner combination was considered to pass this evaluation.

The results of numerous tests embodying the invention are summarized in the following table.

EXAM	SHELL MATERIAL	LINER	ADHESIVE	RESULTS
1	33-400 Phenolic	Norton PT-240 foamed urethane	National 34-9001 urethane	Passed autoclave, 121°C, 30 min. 5 cycles
2	33-400 polypropylene flame treated inside	Corona treated F-217 foamed polyethylene	National 34-9001 urethane	Passed autoclave, one cycle
3	38-430 Phenolic	14B SBR rubber, Armstrong Rubber	National 34-9001 urethane	Passed five cycles in autoclave
4	38-430 Phenolic	Himont 11223-67-1 propylene copolymer	Bostic 9601 urethane	Passed five cycles in autoclave
5	33-400 Phenolic	14B SBR rubber	Bostic 9601-FC urethane	Passed five cycles in autoclave
6	38-400 Melamine	North American Reisse silicone rubber	General Electric R-116 silicone	Passed two cycles in autoclave

It can thus be seen that there has been provided a closure which effectively can be used for hot fill and retortable applications such as food products wherein there is a minimal modification required in making the closure with a liner; wherein there is no need for post-curing equipment; and wherein there is no additional space or labor above that needed for conventional lining operations.

Claims

1. A plastic closure for sealing containers which have been filled with contents that are hot or which are to be autoclaved comprising
   said plastic closure having a base wall and a peripheral skirt,
   said skirt having means thereon for engaging means on a container,
   said base wall of said closure having an inner surface,
   a liner on said inner surface,
   a reactive hot melt adhesive bonding said liner to said inner surface,
   said reactive hot melt adhesive comprising a reactive hot melt adhesive which has become cross-linked after the liner is applied and cured to bond the liner to the inner surface of the base wall of the closure such that the liner will withstand and resist deformation under vacuum caused by cooling of the hot contents in a container or caused by retorting the contents of a container and subsequent cooling.

2. The closure set forth in claim 1 wherein said hot melt adhesive comprises a cross-linkable adhesive selected from the group consisting of polyurethane and silicone.

3. The closure set forth in any one of claims 1 and 2 wherein the closure is made of polypropylene.

4. The closure set forth in any one of claims 1 and 2 wherein the closure is made of phenolic.

5. The closure set forth in any one of claims 1-4 wherein the liner comprises a liner selected from the group consisting of an ethylene, propylede, α-olefin copolymers and SBR rubber.

6. The method of making a plastic closure for sealing containers which have been filled with contents that are hot or which are to be autoclaved comprising

forming a plastic closure having a base wall and a peripheral skirt for engaging a container,

providing a liner on the inner surface of the base wall of the closure,

providing a reactive hot melt adhesive between the liner and the inner surface, said reactive hot melt adhesive comprising a reactive hot melt adhesive which can be crosslinked after the liner is applied and cured,

curing the liner to bond the liner to the inner surface of the base wall of the closure such that the liner will withstand and resist deformation under vacuum caused by cooling of the hot contents in a container or caused by retorting the contents of a container and subsequent cooling.

7. The method set forth in claim 6 wherein said hot melt adhesive comprises a cross-linkable adhesive selected from the group consisting of polyurethane and silicone.

8. The method set forth in any one of claims 6 and 7 wherein the closure is made of polypropylene.

9. The method set forth in any one of claims 6 and 7 wherein the closure is made of phenolic.

10. The method set forth in any one of claims 6-9 wherein the liner comprises a liner selected from the group consisting of an ethylene, propylene, α-olefin copolymers and SBR rubber.

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EUROPEAN SEARCH REPORT

Application Number
EP 94 30 9708

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
A	US-A-4 666 052 (OU-YANG) * column 2, line 63 - column 3, line 48; figures 1-5 *	1	B65D53/04
A	US-A-4 917 949 (YOUSIF) * column 5, line 60 - column 7, line 26; figures 1-3 *	1	
A,D	US-A-4 280 864 (BROMBERG) * column 1, line 64 - column 2, line 7; claim 1; figures 3-8 *	1	
			TECHNICAL FIELDS SEARCHED (Int.Cl.6) B65D

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	4 April 1995	Berrington, N

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
A : technological background
O : non-written disclosure
P : intermediate document

T : theory or principle underlying the invention
E : earlier patent document, but published on, or after the filing date
D : document cited in the application
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